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DATE 10 March 1964

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ABLATION SHIELD DEVELOPMENT TESTING - ADHESIVE EVALUATION AND ELEVATED TEMPERATURE PROPERTIES

REPORT A472 SERIAL NO. 20

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STRUCTURES LABORATORY FINAL REPORT

ABLATION SHIELD DEVELOPMENT TESTING - ADHESIVE EVALUATION AND ELEVATED TEMPERATURE PROPERTIES

ABSTRACT

Reduction in weight of component parts of space vehicles is desirable in order that a larger proportion of the "payload" might be devoted to scientific and life-sustaining equipment to extend the mission capabilities of space vehicles. Thus, a four phase development program was initiated to determine the best materials and fabrication techniques for fabrication of a light weight ablation shield. The proposed design is the result of studies conducted in an effort to reduce the weight of advanced space vehicles. It was the purpose of this phase of the development program to evaluate various adhesives and adhesive curing cycles for adhesive bonding of beryllium.

Surfaces of beryllium finger panels were processed for adhesive bonding by the optimum surface preparation method established by a previous test of this development program. Various film adhesives and bonding methods for each adhesive were then used in bonding the beryllium finger panels together. After bond line curing the bonded finger panels were machined into individual lap shear tension test specimens. Test specimens were subjected to a series of tests for adhesive shear strength at room and elevated temperatures.

Although the lap shear strength of HT-424 film adhesive manufactured by the Bloomingdale Rubber Company was lower than other adhesives tested at room temperature and 500F its greater strength at 650F and 800F makes it the most desireable adhesive for use in bonding applications involving beryllium and high temperature environments.

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DISTRIBUTION: T. P. Brooks, W. H. Gray, M. S. Hochberg, C. Wadleigh, Eng. Library

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1. OBJECT

The object of this phase of the space vehicle ablation shield development program was to evaluate three types of adhesive for use in adhesive bonding of beryllium. Two different curing methods for each adhesive were also to be evaluated. The best adhesive and adhesive curing method, as determined by test results, will then be used in succeeding phases of the ablation shield development program.

2. CASE HISTORY

Additional scientific and life sustaining equipment for extended mission capabilities of advanced space vehicle designs has resulted in an increase in weight of the space vehicle. A means of compensating for a portion of this additional weight is a proposed lightweight re-entry shield consisting of an optimum ablative material suitably bonded to a beryllium back-up structure. A four phase development/evaluation program to establish design and fabrication criteria for such a shield was initiated. The initial phase of the development/evaluation program (TR 052-051.03.01) established an optimum surface preparation method for adhesive bonding of beryllium. It was the purpose of this phase of the development/evaluation program to determine the best adhesive and adhesive curing cycle for adhesive bonding of beryllium.

Testing was conducted by the Systems Laboratory during the period 5 February 1962 through 3 March 1962.

3. SPECIMEN PREPARATION

Sixty beryllium finger panels were machined per dimensions as shown in Figure 1, page 6, from QMV-200-A press sintered block beryllium. All machining was performed by the Brush Beryllium Company, prior to shipment of the finger panels to McDonnell. Machined beryllium panels, rather than rolled beryllium sheet stock panels were used in order to closely simulate conditions which would be encountered in actual fabrication. Finger panels were divided into six groups, each group containing ten panels.

The beryllium finger panels were processed for adhesive bonding, using the optimum surface preparation method established by TR 052-051.03.01. See Bonding Method "G", Step 1 in Table 1, page 12, for a brief outline of this surface preparation process. Upon completion of the surface preparation processes the finger panels were placed between clean, lint-free cheesecloth and wrapped in wax-free kraft paper. Bonding operations were performed within twenty-four hours after completion of the surface preparation processes.

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4. SPECIMEN BONDING

The three adhesives and the two curing cycles for each adhesive which were evaluated by this test program are outlined in Table 1, page 12. Each individual adhesive and its respective curing cycle was assigned a code letter ("G" through "M" (omitting "I") as shown in Table 1 for purposes of specimen identification after bonding operations.

The adhesives which were evaluated and their respective manufacturer's are:

(a) HT-424 Film Adhesive,.015 thick, 0.13-0.14 lbs/ft^2, Bloomingdale Rubber Company, Aberdeen, Maryland

(b) AF-107 film adhesive,.015 thick and EC-1639 primer 20% solids, Minnesota Mining and Manufacturing Company, 6411 Randolph, Los Angeles, California.

(c) Aerobond 430, .015 thick, Adhesive Engineering Company, 1411 Industrial Road, San Carlos, California.

HT-424 adhesive was stored at 0°F or less and the remaining two types of adhesives were stored at 35°-40°F. At the time of usage, the three types of adhesives were not more than thirty days old.

An amount of adhesive sufficient for bonding a set of finger panels and corresponding to the type required by the bonding method being evaluated was removed from the refrigerated roll approximately two hours prior to installation in the bond line to allow the adhesive to return to ambient temperature.

Bonding fixtures of the type as shown in Figures 2, 3, 4, and 5 on pages 7, and 8, were used in bonding the beryllium finger panels together. Prior to the bonding operations a set of springs for each bonding fixture was calibrated so that at a given spring compression deflection a pressure of 30 psi would be applied to the bond line area.

One finger panel was mounted in each of five bonding fixtures and secured in place as shown in Figure 2, page 7. A strip of the film adhesive being evaluated was placed over the finger panel as shown in Figure 3, page 7. The remaining five panels were mounted in the bonding fixtures and secured in place as shown in Figure 4, page 8. A strip of rubber was placed over the bond line area to provide for a constant even pressure over the bond line. The clamping block, springs and flanged nuts were then installed as shown in Figure 5, page 8. A thermocouple for monitoring bond line temperature was installed between the upper beryllium panels and the rubber strip. The springs were then compressed until a previously calibrated spring deflection which produced a 30 psi bond line pressure was attained.

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4. SPECIMEN BONDING (cont'd.)

The film adhesive being evaluated was cured per one of the respective curing methods, as outlined in Table 1, page 12. After completion of the bond line cure cycle the bonding fixtures were removed from the oven and allowed to cool to 150F before releasing the spring pressure. Bond line temperature of the five fixtures was monitored by the thermocouple setup as shown in Figure 6, page 9 .

After cooling, each individual finger of the bonded panels was marked with the code letter assigned to the bonding method which was being evaluated. A number (1 through 20) was then assigned to each individual finger of the bonded panels. After identification of the individual fingers was completed, the bonded finger panels were sent to the beryllium machining facility and machined into individual lap shear tension test specimens as shown in Figure 7, page 9 .

5. TEST SETUP

Setups for testing the bond line strength in lap shear at room and elevated temperatures were mounted in the 5,000 pound Baldwin tensile test machine. Standard grips, as shown in Figure 8, page 10, were the only items required for room temperature testing. For high temperature testing the linkage as shown in Figure 8 was used to prevent the grips from being in the heated area. Radiant heat lamps mounted as shown in Figure 8 were positioned vertically so that the heat concentration would be centered on the test area. Power source for and control of the radiant heat lamps was provided by a Research Incorporated, Model 6233 Ignitron. A Leeds and Northrup indicating pyrometer for monitoring the outer surface temperature of the test specimen was included in the test setup. Test specimen outer surface temperature, rather than actual bond line temperature was monitored to allow for a simpler and faster temperature monitoring setup. A comparative test between the bond line temperature and the outer surface temperature was conducted and indicated no significant temperature difference.

5. TEST PROCEDURES

Lap shear tension testing was conducted at room temperature and at elevated temperatures of 500F, 650F, and 800F. A total of five specimens were tested at each of the four required test temperatures.

Loading rate for all test temperatures was 600-700 lbs/min. Heating rate for the elevated temperature tests was 100F/min. Followed by a ten minute soak at the specified temperature.

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6. TEST PROCEDURES (cont'd)

Bond line thicknesses of all specimens were measured prior to testing and are recorded in Tables 2 through 7, pages 13 through 18, respectively. Bond line areas for the various specimens are also recorded in the previously mentioned tables.

7. TEST RESULTS

Bond line lap shear tension test results for the various groups of test specimens are presented in Tables 2 through 7 on pages 13, through 18, respectively. Failing loads in pounds, failing stress in psi, failing stress levels in psi and the nature of failure are included in these tables. A graph comparing the strength versus temperature curves of the six bonding methods evaluated by this test program is shown in Figure 9, page 11.

8. CONCLUSIONS

The best film adhesive and adhesive curing method, to be used in bonding applications involving beryllium and high temperatures (650F - 800F), is HT-424 film adhesive and bonding method "G", respectively. This selection is based on the comparative lap shear strengths obtained at the test levels previously mentioned. Although the strength of HT-424 at room temperature and 500F is lower than other adhesives tested its greater strength at 650F and 800F temperatures was the predominant feature which was considered in the final analysis. Bonding method "G" rather than "H" was selected on the basis of its simpler curing procedure.

LIST OF EQUIPMENT AND INSTRUMENTS

Equipment and instruments used in this test are listed below. Applicable calibration records are available for inspection.

Item	Manufacturer and Model Number	Serial or Laboratory Number
Oven	Grieve-Hendry Co. Inc. Model HX500	MAC 40255-51
Indicating Pyrometer	Leeds and Northrup	MAC 3709
5,000 LB Tensile Test Machine	Baldwin-Tate-Emery Model P.T.E. 27	USN 800879
Ignitron	Research Inc. Model 6231	MAC 33506-1

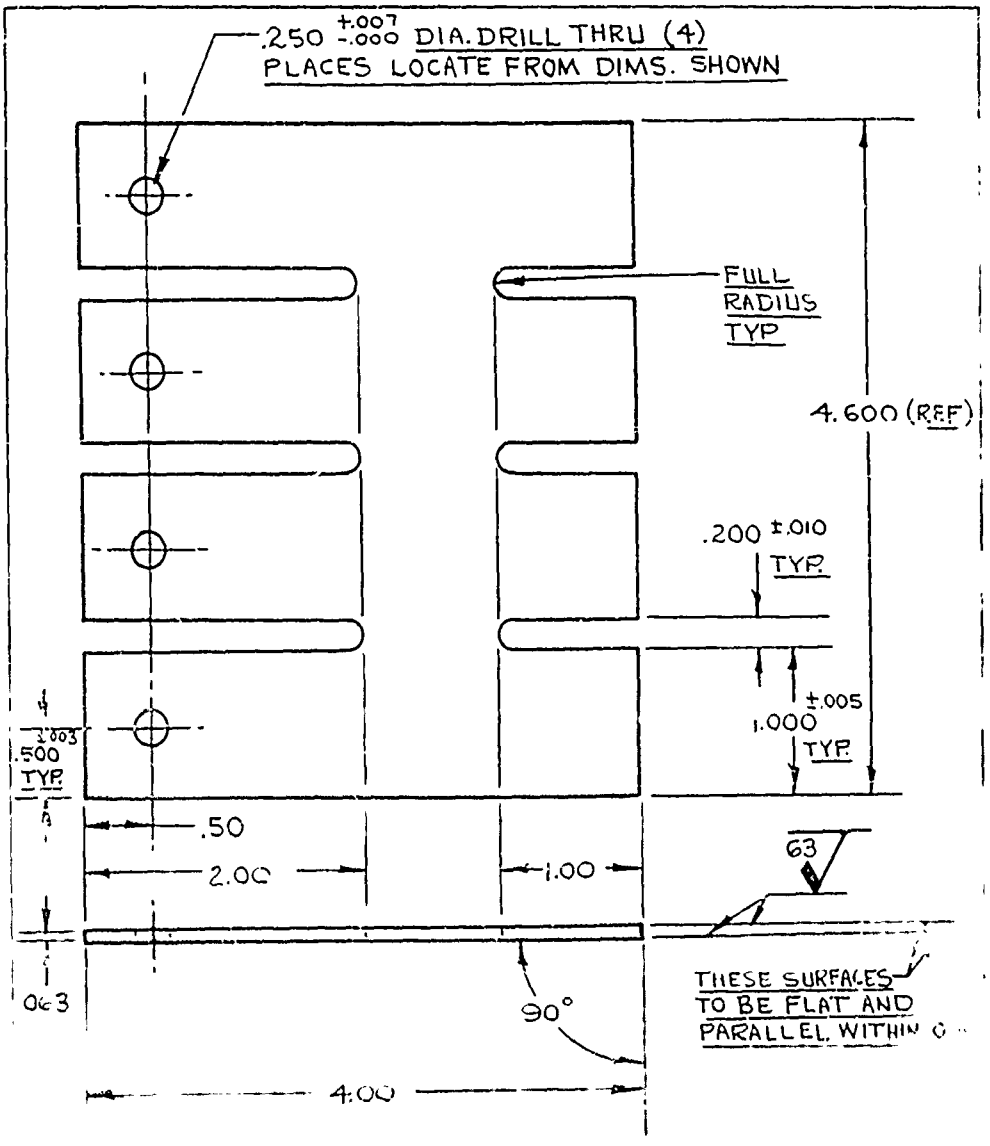


FIGURE NUMBER 1
BERYLLIUM FINGER PANELS ~
DIMENSIONAL REQUIREMENTS

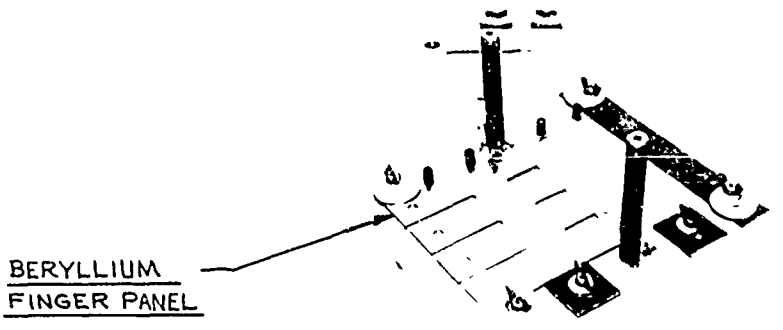


FIGURE NUMBER 2
BOND FIXTURE LOADING ~ STEP 1
(PHOTO NO. D4E245807)

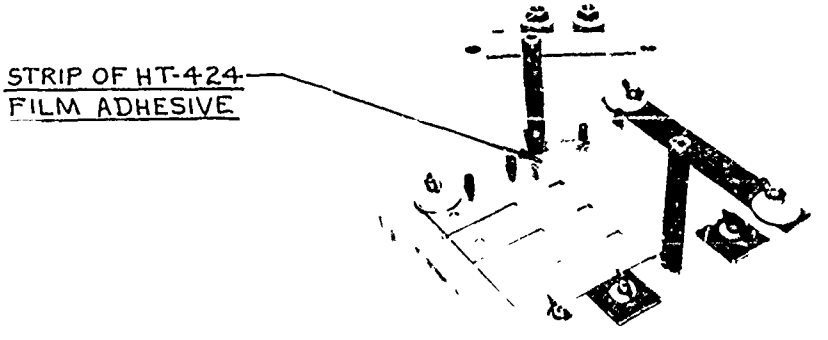


FIGURE NUMBER 3
BOND FIXTURE LOADING ~ STEP 2
(PHOTO NO. D4E245805)

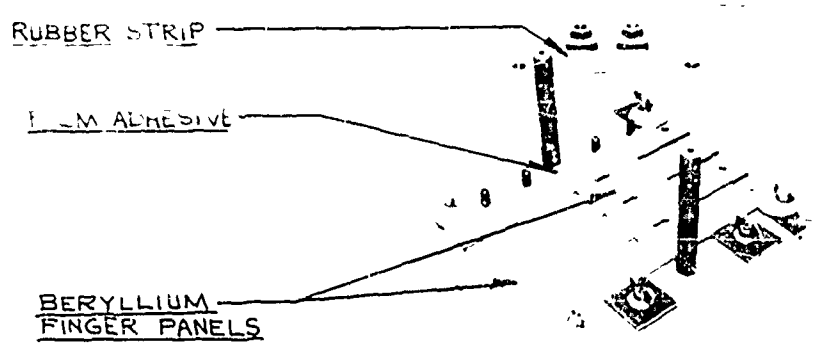


FIGURE NUMBER 4
BOND FIXTURE LOADING ~ STEP 3
(PHOTO NO. D4E 245806)

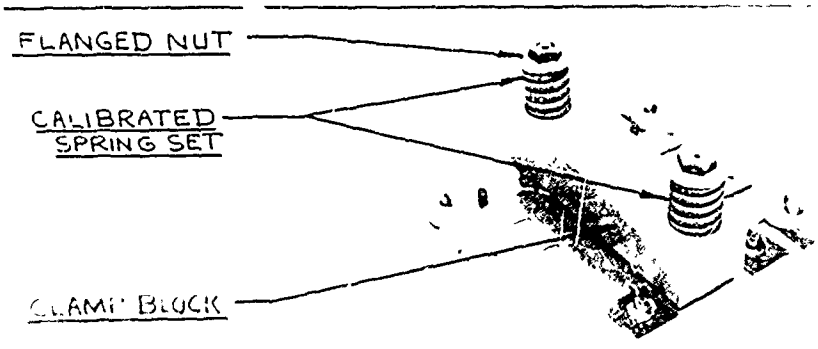


FIGURE NUMBER 5
BOND FIXTURE LOADING ~ STEP 4
(PHOTO NO. D4E 245804)

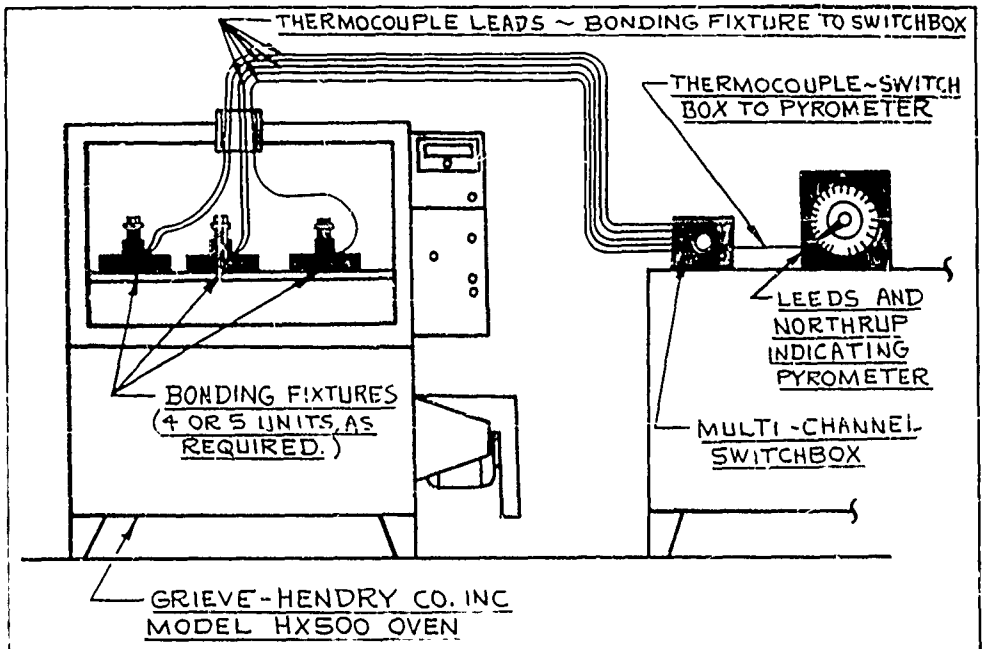


FIGURE NUMBER 6
BOND LINE TEMPERATURE MONITORING SET UP

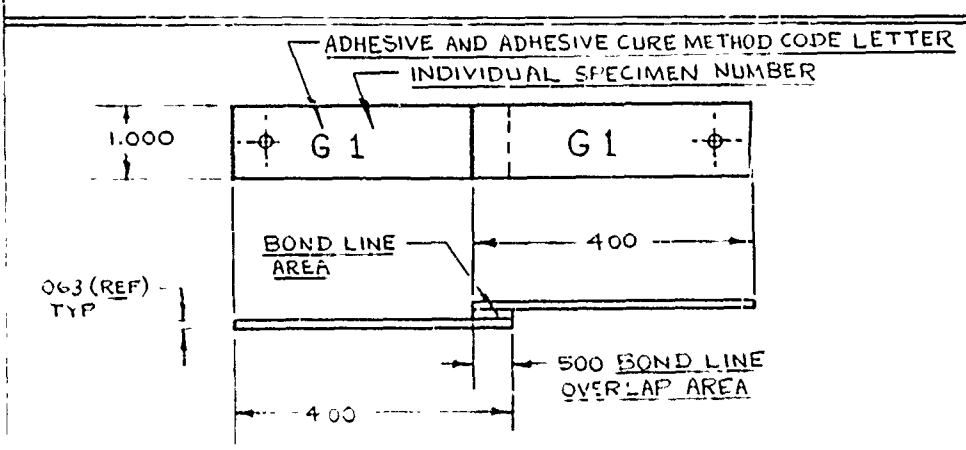


FIGURE NUMBER 7
BOND LINE LAP SHEAR TENSION TEST SPECIMEN

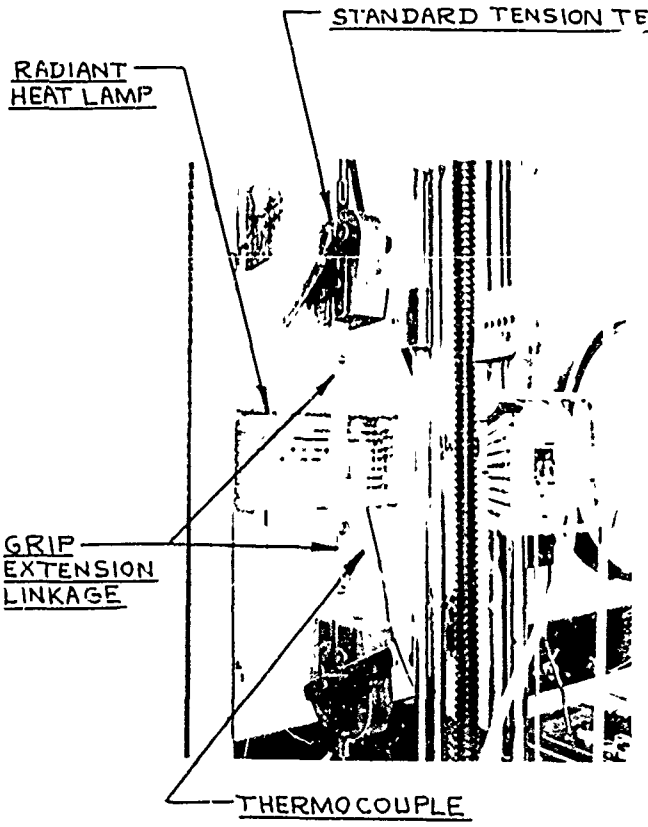
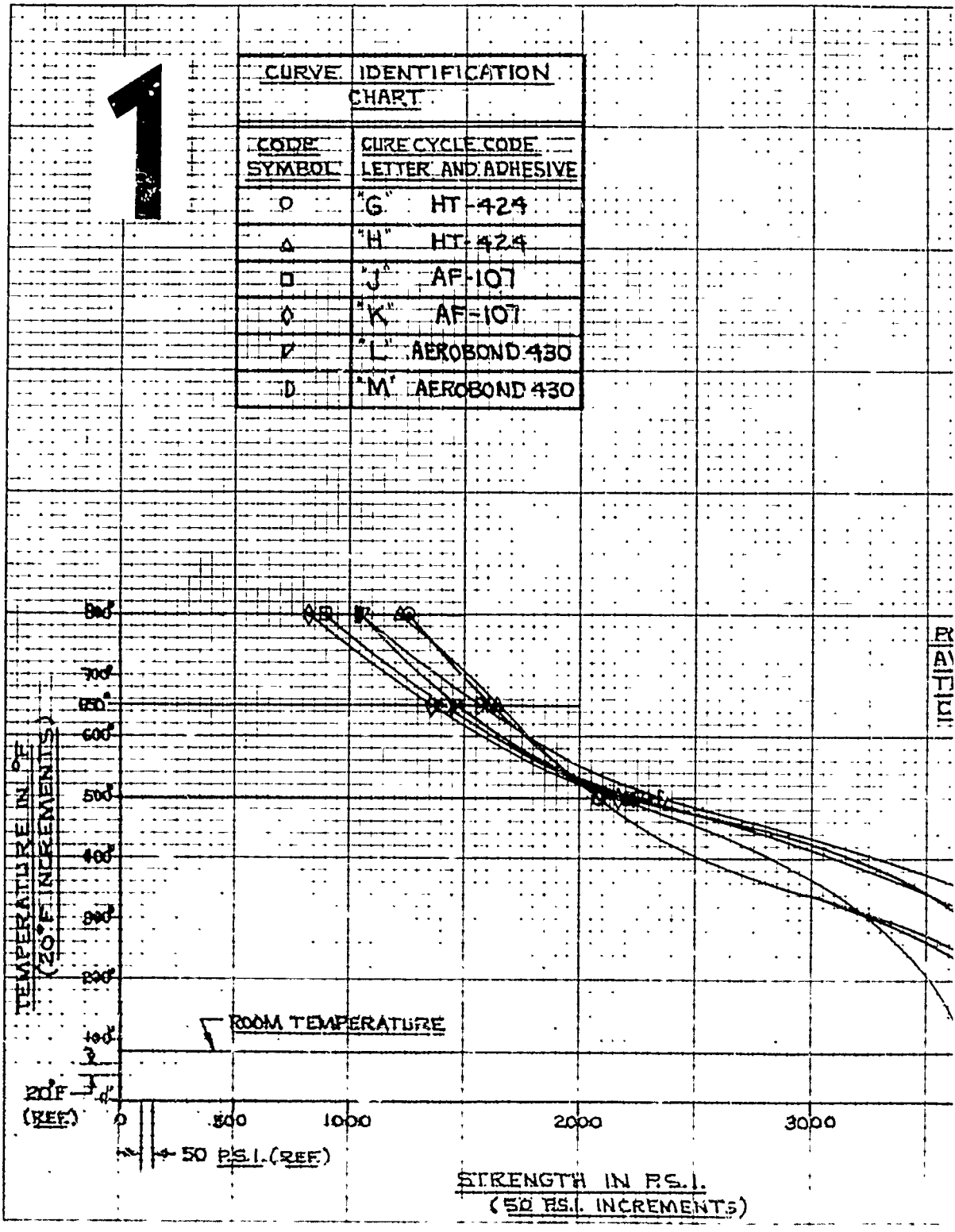


FIGURE NUMBER 8

LAP SHEAR TENSION TEST SETUP IN THE 5,000 LB. BALDWIN TENSILE TEST MACHINE

(PHOTO NO. D4E 245800)


1
CURVE IDENTIFICATION CHART
CODE SYMBOL
CURE CYCLE CODE LETTER AND ADHESIVE
○ "G" HT-424
△ "H" HT-424
□ "J" AF-107
◇ "K" AF-107
▽ "L" AEROBOND 430
▷ "M" AEROBOND 430
TEMPERATURE IN °F (20°F INCREMENTS)
800°
700°
600°
500°
400°
300°
200°
100°
ROOM TEMPERATURE
20°F (REF.)
0
500
1000
2000
3000
50 P.S.I. (REF.)
STRENGTH IN P.S.I. (50 P.S.I. INCREMENTS)

ON

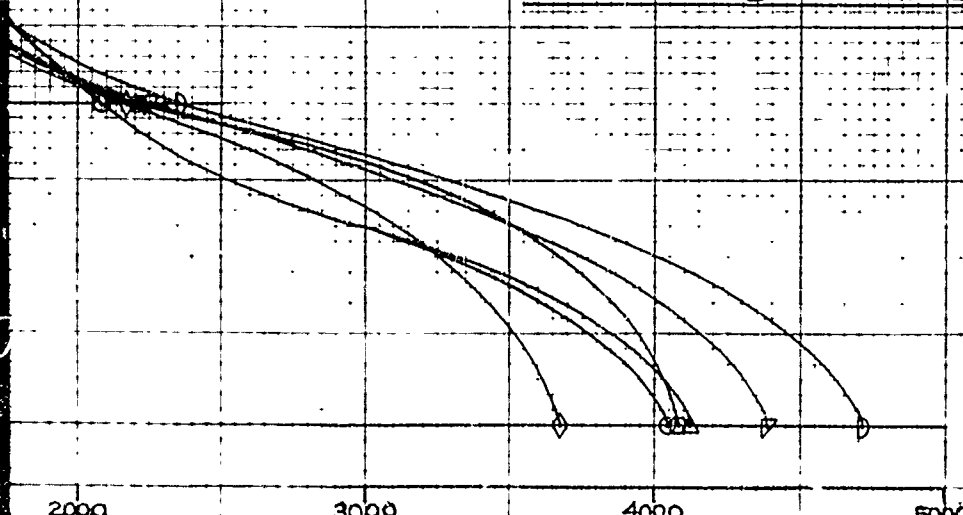
ESIVE

430

430

2

POINTS ON THIS GRAPH REPRESENT AVERAGE FAILING STRESS IN P.S.I. FOR THE VARIOUS ADHESIVES AND ADHESIVE CURING METHODS EVALUATED



2000 3000 4000 5000

NGTH IN P.S.I.
P.S.I. INCREMENTS)

FIGURE NUMBER 9

COMPARISON GRAPH - STRENGTH VS. TEMP. - CURVES OF THE VARIOUS ADHESIVES AND ADHESIVE CURING METHODS EVALUATED.

ADHESIVE AND BONDING METHODS EVALUATED BY THIS TEST PROGRAM

ADHESIVE	BONDING METHOD	STEP BY STEP PROCEDURE
HT-424	G	1.) PREPARE BERYLLIUM SURFACES TO BE BONDED PER THE FOLLOWING PROCEDURES; a) VAPOR DEGREASE PER MAC P.S. 12020, b) LIQUID HONE USING BURR-AL 220 GRIT, c) ALKALINE CLEAN PER MAC P.S. 12030 TYPE I WITH NO CURRENT. 2.) REMOVE A SUFFICIENT AMOUNT OF ADHESIVE FROM THE REFRIGERATED ROLL, WRAP IN MYLAR AND ALLOW TO RETURN TO AMBIENT TEMPERATURE. 3.) CONNECT THERMOCOUPLE FROM THE INDICATING PYROMETER TO THE (5) CHANNEL SWITCHBOX. CONNECT (5) THERMOCOUPLE LEADS TO THE SWITCHBOX. 4.) INSTALL BERYLLIUM PANELS, ADHESIVE AND THERMOCOUPLES IN THE BONDING FIXTURES. 5.) APPLY 30 P.S.I. PRESSURE TO THE BOND LINE. 6.) CURE THE BOND LINE PER THE FOLLOWING PROCEDURES. a) PLACE FIXTURES IN A 90°F PREHEATED OVEN. b) COMMENCE HEATING AT A 4°F/MIN. HEATING RATE FOR 60 MINS. (4°F X 60 = 240°F, 240°F + 90°F = 330°F) c) HOLD 330° ±5°F FOR 120 MINUTES. d) REMOVE FIXTURES FROM OVEN AND ALLOW TO COOL TO 150°F BEFORE RELEASING SPRING PRESSURE. e) REMOVE PANELS FROM FIXTURE AND IDENTIFY WITH CODE LETTER "G."
HT-424	H	1.) SEE STEPS 1, 2, 3, 4 AND 5 AS NOTED IN METHOD "G" ABOVE. 2.) CURE THE BOND LINE PER THE FOLLOWING PROCEDURES. a) PLACE FIXTURES IN A 90°F PREHEATED OVEN. b) COMMENCE HEATING AT A 4°F/MIN. HEATING RATE FOR 60 MINS. (4°F X 60 = 240°F, 240°F + 90°F = 330°F) c) HOLD 330° ±5°F FOR 60 MINUTES. d) RAISE TEMPERATURE AT A 7°F/MIN. HEATING RATE FOR 10 MINS. (7°F X 10 = 70°F, 330°F + 70°F = 400°F) e) HOLD 400° ±5°F FOR 60 MINUTES. f) REMOVE FIXTURES FROM OVEN AND ALLOW TO COOL TO 150°F BEFORE RELEASING SPRING PRESSURE. g) REMOVE PANELS FROM FIXTURE AND IDENTIFY WITH CODE LETTER "H"
EC 1639 AF 107	J	1) SEE STEPS 1 AND 2 AS NOTED IN METHOD "G" ABOVE. 2.) APPLY A .0005 FILM THICKNESS OF EC1639 TO THE AREAS OF THE BERYLLIUM PANELS TO BE BONDED. AIR DRY 30 MINS. FORCE DRY 30 MINS. AT 325° ±5°F. 3) SEE STEPS 3, 4, AND 5 AS NOTED IN METHOD "G" ABOVE.

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HT-424	H	1.) SEE STEPS 1, 2, 3, 4 AND 5 AS NOTED IN METHOD "G" ABOVE. 2.) CURE THE BOND LINE PER THE FOLLOWING PROCEDURES. a) PLACE FIXTURES IN A 90°F PREHEATED OVEN b) COMMENCE HEATING AT A 4°F/MIN. HEATING RATE FOR 60 MINS. (4°F X 60 = 240°F, 240°F + 90°F = 330°F) c) HOLD 330° ±5°F FOR 60 MINUTES d) RAISE TEMPERATURE AT A 7°F/MIN. HEATING RATE FOR 10 MINS. (7°F X 10 = 70°F, 330°F + 70°F = 400°F) e) HOLD 400° ±5°F FOR 60 MINUTES. f) REMOVE FIXTURES FROM OVEN AND ALLOW TO COOL TO 150°F BEFORE RELEASING SPRING PRESSURE. g) REMOVE PANELS FROM FIXTURE AND IDENTIFY WITH CODE LETTER "H".
EC 1639 AF 107	J	1.) SEE STEPS 1 AND 2 AS NOTED IN METHOD "G" ABOVE. 2.) APPLY A .0005 FILM THICKNESS OF EC1639 TO THE AREAS OF THE BERYLLIUM PANELS TO BE BONDED. AIR DRY 30 MINS. FORCE DRY 30 MINS. AT 325°±5°F. 3.) SEE STEPS 3, 4 AND 5 AS NOTED IN METHOD "G" ABOVE. 4.) CURE THE BOND LINE PER THE FOLLOWING PROCEDURE, a) PLACE FIXTURES IN A 80°F PREHEATED OVEN. b) COMMENCE HEATING AT A 3°F/MIN. HEATING RATE FOR 90 MINS. (3°F X 90 = 270°F, 270°F + 80°F = 350°F c) HOLD 350°±5°F FOR 60 MINS. d) REMOVE FIXTURES FROM OVEN AND ALLOW TO COOL TO 150°F BEFORE RELEASING SPRING PRESSURE. e) REMOVE PANELS FROM FIXTURE AND IDENTIFY WITH CODE LETTER "J."
EC1639 AF107	K	1.) SEE STEPS 1, 2 AND 3 AS NOTED IN METHOD "J" ABOVE. 2.) CURE THE BOND LINE PER THE FOLLOWING PROCEDURE; a) PLACE FIXTURE IN A 80°F PREHEATED OVEN. b) COMMENCE HEATING AT A 3°F/MIN. HEATING RATE FOR 55 MINS. (3°F X 55 = 165°F, 165°F + 80°F = 245°F) c) CONTINUE HEATING AT A ½°F/MIN. HEATING RATE FOR 90 MINS. (½°F X 90 = 45°F, 45°F + 245°F = 290°F) d) CONTINUE HEAT [illegible] A 3°F/MIN. HEATING RATE FOR 20 MINS. (3°F X 20 = 60°F, 60°F + 290°F = 350°F) e) HOLD 350°±5°F FOR 60 MINS. f) REMOVE FIXTURES FROM OVEN AND ALLOW TO COOL TO 150°F BEFORE RELEASING SPRING PRESSURE. g) REMOVE PANELS FROM FIXTURE AND IDENTIFY WITH CODE LETTER "K".
AEROBOND 430	L	1.) SEE STEPS 1, 2, 3, 4 AND 5 AS NOTED IN METHOD "G" ABOVE. 2.) CURE THE BOND LINE AS FOLLOWS, a) PLACE FIXTURES IN A 70°F PREHEATED OVEN. b) COMMENCE HEATING AT A 13°F/MIN. HEATING RATE FOR 20 MINS. (13°F X 20 = 260°F, 260°F + 70°F = 330°F), c) HOLD 330°±5°F FOR 60 MINS., d) REMOVE FIXTURES FROM OVEN AND ALLOW TO COOL TO 150°F BEFORE RELEASING SPRING PRESSURE, e) REMOVE PANELS FROM FIXTURE AND IDENTIFY WITH CODE LETTER "L"
AEROBOND 430	M	1.) SEE STEPS 1, 2, 3, 4 AND 5 AS NOTED IN METHOD "G" ABOVE. 2.) CURE THE BOND LINE AS FOLLOWS; a) PLACE FIXTURES IN A 80°F PREHEATED OVEN b) COMMENCE HEATING AT A 5°F/MIN. HEATING RATE FOR 50 MINS. (5°F X 50 = 250°F, 250°F + 80°F = 330°F), c) HOLD 330°±5°F FOR 60 MINS., d) REMOVE FIXTURES FROM OVEN AND ALLOW TO COOL TO 150°F BEFORE RELEASING SPRING PRESSURE, e) REMOVE PANELS FROM FIXTURE AND IDENTIFY WITH CODE LETTER "M".

TABLE NUMBER 1

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SPECIMEN NUMBER	BOND LINE AREA (IN.²)	BOND LINE THICKNESS	TEMPERATURE REQUIREMENTS	FAILING LOAD (POUNDS)	FAILING STRESS (P.S.I.)	FAILING STRESS LEVELS (P.S.I.)	NATURE OF FAILURE CODE LETTERS Be: BERYLLIUM A: ADHESIVE C: COHESIVE
G1	.375	.006	ROOM TEMPERATURE	1530	4080	MAX 4280 AVE. 4044 MIN. 3896	100% Be
G2	.375	.006		1460	3896		100% Be
G3	.375	.006		1605	4280		100% C
G4	.375	.007		1470	3920		100% Be
G5	500	.007		1740	3480		95% Be 5% C
G6	.500	.006	500°F AFTER 10 MINUTES AT 500°F	1060	2120	MAX 2224 AVE. 2078 MIN. 1890	100% C
G7	.500	.006		1015	2030		100% C
G8	.500	.006		945	1890		100% C
G9	.500	.007		1065	2130		100% C
G10	.500	.007		1112	2224		100% C
G11	.500	.007	650°F AFTER 10 MINUTES AT 650°F	862	1726	MAX 1726 AVE. 1620 MIN. 1508	100% C
G12	.500	.007		787	1574		100% C
G13	.500	.006		754	1508		100% C
G14	.500	.006		836	1672		100% C
G15	.500	.006		812	1624		100% C
G16	.500	.007	800°F AFTER 10 MINUTES AT 800°F	580	1160	MAX. 1312 AVE. 1263 MIN. 1160	100% C
G17	.500	.007		617	1234		100% C
G18	.500	.006		652	1304		100% C
G19	.500	.006		656	1312		100% C
G20	.500	006		653	1306		100% C

TABLE NUMBER 2

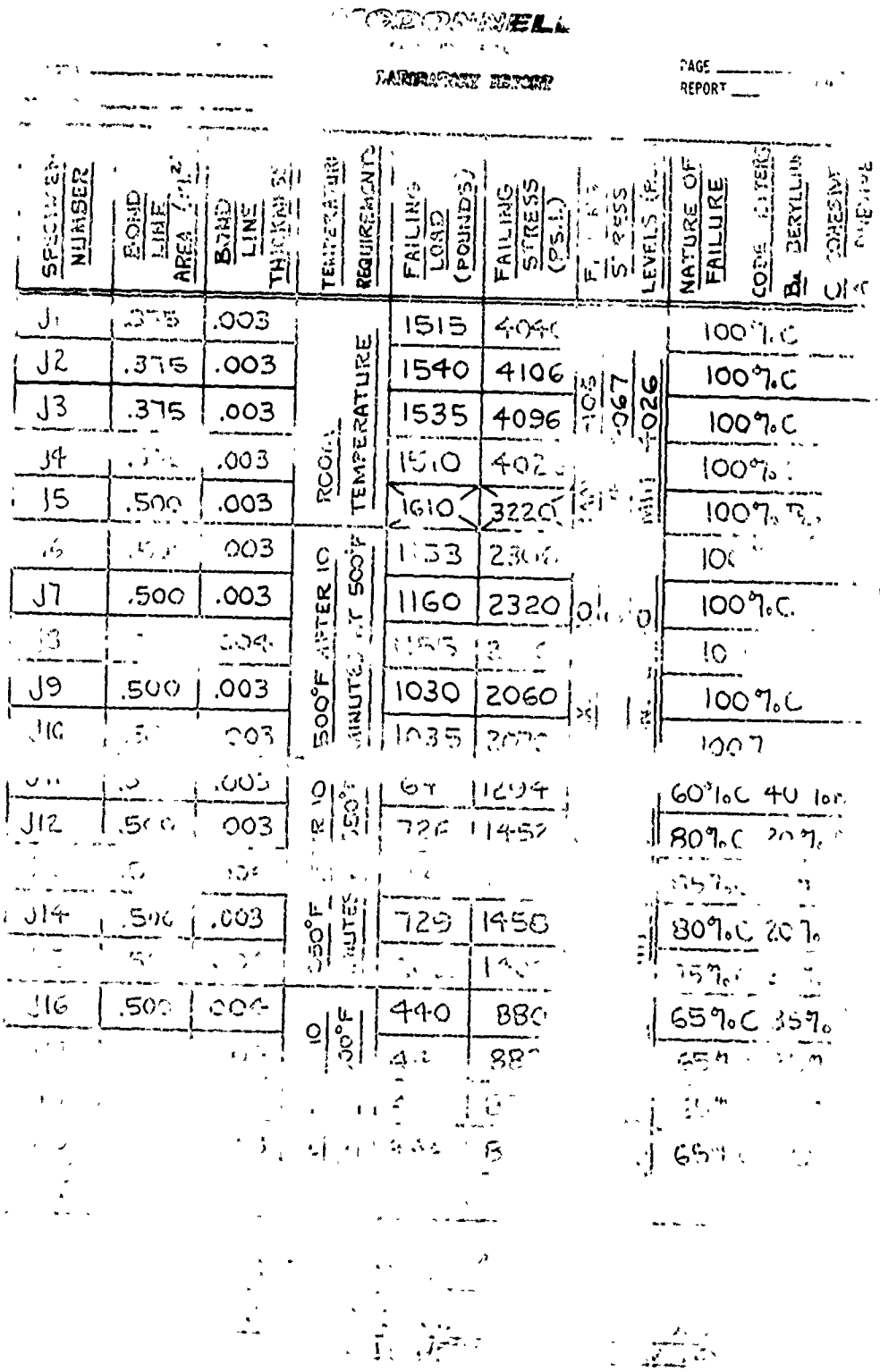
TEST RESULTS ~ BONDING METHOD "G" ~ HT-424 ADHESIVE

(SPECIMEN G5 NOT INCLUDED IN AVERAGE FOR ROOM TEMP GROUP)

SPECIMEN NUMBER	BOND LINE AREA (IN.²)	BOND LINE THICKNESS	TEMPERATURE REQUIREMENTS	FAILING LOAD (POUNDS)	FAILING STRESS (P.S.I.)	FAILING STRESS LEVELS (P.S.I.)	NATURE OF FAILURE CODE LETTERS Bₑ - BERYLLIUM A: ADHESIVE C: COHESIVE
H1	.375	.008	ROOM TEMPERATURE	1570	4186	MAX. 4186 AVE. 4126 MIN. 4026	90%Bₑ 10%C
H2	.375	.006		1555	4146		100%Bₑ
H3	.375	.005		1510	4026		100%Bₑ
H4	.375	.006		1555	4146		100%C
H5	.500	.006		1650	3300		100%Bₑ
H6	.500	.006	500°F AFTER 10 MINUTES AT 500°F	1060	2120	MAX. 2130 AVE. 2070 MIN 1960	100%C
H7	.500	.006		1065	2130		100%C
H8	.500	.007		1010	2020		100%C
H9	.500	.007		980	1960		100%C
H10	.500	.006		1060	2120		100%C
H11	.500	.006	650°F AFTER 10 MINUTES AT 650°F	786	1572	MAX. 1782 AVE 1646 MIN. 1520	100%C
H12	.500	.006		760	1520		100%C
H13	.500	.007		839	1678		100%C
H14	.500	.007		891	1782		100%C
H15	.500	.006		838	1676		100%C
H16	.500	.007	800°F AFTER 10 MINUTES AT 800°F	597	1194	MAX 1260 AVE 1222 MIN 1182	100%C
H17	500	.006		609	1218		100%C
H18	.500	006		627	1254		100%C
H19	500	.006		630	1260		100%C
H20	.500	.007		591	1182		100%C

TABLE NUMBER 3

TEST RESULTS ~ BONDING METHOD "H" ~ HT-424 ADHESIVE
(SPECIMEN H5 NOT INCLUDED IN AVERAGE FOR ROOM TEMP GROUP



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SPECIMEN NUMBER	BOND LINE AREA (SQ. IN.)	BOND LINE THICKNESS	TEMPERATURE REQUIREMENTS	FAILING LOAD (POUNDS)	FAILING STRESS (P.S.I.)	FAILING STRESS LEVELS (P.S.I.)	NATURE OF FAILURE	CODE LETTERS	% BERYLLIUM	% CORESIN
J1	.375	.003	ROOM TEMPERATURE	1515	4040		100% C			
J2	.375	.003		1540	4106		100% C			
J3	.375	.003		1535	4096	1026 1067 1105	100% C			
J4	.375	.003		1510	4027		100% C			
J5	.500	.003		1610	3220		100% C			
J6	.500	.003	500°F AFTER 10 MINUTES AT 500°F	1153	2306		100% C			
J7	.500	.003		1160	2320	0	100% C			
J8	.500	.003		1155	2310		100% C			
J9	.500	.003		1030	2060		100% C			
J10	.500	.003		1035	2070		100% C			
J11	.500	.003	450°F FOR 10 MINUTES	671	1294		60% C	40		
J12	.500	.003		726	1452		80% C	20		
J13	.500	.003					85% C			
J14	.500	.003		729	1458		80% C	20		
J15	.500	.003			1452		75% C			
J16	.500	.003	400°F FOR 10 MINUTES	440	880		65% C	35		
J17	.500	.003		441	882		65% C			
J18	.500	.003								
J19	.500	.003					65% C			

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ST. LOUIS, MISSOURI

DATE _____

REVISED _____

REVISED _____

LABORATORY REPORT

SPECIMEN NUMBER	BOND LINE AREA (IN.²)	BOND LINE THICKNESS	TEMPERATURE REQUIREMENTS	FAILING LOAD (POUNDS)	FAILING STRESS (P.S.I.)	FAILING STRESS LEVELS (P.S.I.)	NATURE OF FAILURE CODE LETTERS Be = BERYLLIUM C = COHESIVE A = ADHESIVE
K1	.375	.005	ROOM TEMPERATURE	1350	3600	MAX. 3826 AVE. 3676 MIN. 3560	85%C 15%A
K2	.375	.004		1335	3560		80%C 20%A
K3	.375	.004		1395	3720		90%C 10%A
K4	.375	.004		1435	3826		100%C
K5	.500	.004		1425	2850		75%C 25%A
K6	.500	.003	500°F AFTER 10 MINUTES AT 500°F	1077	2154	MAX. 2230 AVE. 2172 MIN. 2140	100%C
K7	.500	.003		1072	2144		100%C
K8	.500	.003		1070	2140		100%C
K9	.500	.004		1115	2230		100%C
K10	.500	.004		1095	2190		100%C
K11	.500	.004	650°F AFTER 10 MINUTES AT 650°F	679	1358	MAX. 1576 AVE. 1365 MIN. 1016	60%C 40%A
K12	.500	.004		788	1576		85%C 15%A
K13	.500	.005		508	1016		40%C 60%A
K14	.500	.005		742	1484		75%C 25%A
K15	.500	.005		695	1390		60%C 40%A
K16	.500	.004	800°F AFTER 10 MINUTES AT 800°F	414	828	MAX. 964 AVE. 823 MIN. 682	60%C 40%A
K17	.500	.005		482	964		70%C 30%A
K18	.500	.005		424	848		60%C 40%A
K19	.500	.004		397	794		55%C 45%A
K20	.500	.004		341	682		40%C 60%A

TABLE NUMBER 5

TEST RESULTS ~ BONDING METHOD "K" ~ AF107 ADHES

(SPECIMEN K5 NOT INCLUDED IN AVERAGE FOR ROOM TEMP. GROUP)

REVISED
REVISED

SPECIMEN NUMBER	BOND LINE AREA (IN.²)	BOND LINE THICKNESS	TEMPERATURE REQUIREMENTS	FAILING LOAD (POUNDS)	FAILING STRESS (P.S.I.)	FAILING STRESS LEVELS (P.S.I.)	NATURE OF FAILURE CODE LETTERS Be=BERYLLIUM C=COHESIVE A=ADHESIVE
L1	.375	.007	ROOM TEMPERATURE	1700	4533	MAX. 4533 AVE. 4383 MIN. 4266	100% Be
L2	.375	.007		1635	4360		100% Be
L3	.375	.007		1640	4373		100% Be
L4	.375	.007		1600	4266		100% Be
L5	.500	.008		1870	3740		100% Be
L6	.500	.007	500°F AFTER 10 MINUTES AT 500°F	1105	2210	MAX. 2230 AVE. 2217 MIN. 2210	100% C
L7	.500	.007		1110	2220		100% C
L8	.500	.007		1125	2250		100% C
L9	.500	.008		1140	2280		100% C
L10	.500	.008		1138	2276		100% C
L11	.500	.007	650°F AFTER 10 MINUTES AT 650°F	733	1466	MAX. 1513 AVE. 1454 MIN. 1328	100% C
L12	.500	.008		742	1484		100% C
L13	.500	.009		664	1328		100% C
L14	.500	.007		738	1476		100% C
L15	.500	.007		759	1518		100% C
L16	.500	.007	800°F AFTER 10 MINUTES AT 800°F	536	1072	MAX. 1098 AVE. 1053 MIN. 946	100% C
L17	.500	.007		473	946		100% C
L18	.500	.007		537	1074		100% C
L19	.500	.007		537	1074		100% C
L20	.500	.007		549	1098		100% C

TABLE NUMBER 6

TEST RESULTS ~ BONDING METHOD "L" ~ AEROBOND [illegible]
(SPECIMEN L5 NOT INCLUDED IN AVERAGE FOR ROOM TEMP. GROUP.)

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LABORATORY REPORT

SPECIMEN NUMBER	BOND LINE AREA (IN.²)	BOND LINE THICKNESS	TEMPERATURE REQUIREMENTS	FAILING LOAD (POUNDS)	FAILING STRESS (P.S.I.)	FAILING STRESS LEVELS (P.S.I.)	NATURE OF FAILURE (CODE LETTERS B=BERYLLIUM C=COHESIVE A=ADHESIVE)
M1	.375	.007	ROOM TEMPERATURE	1830	4880	MAX. 4880 AVE. 4710 MIN. 4440	100% Be
M2	.375	.007		1780	4746		100% Be
M3	.375	.007		1790	4773		100% Be
M4	.375	.008		1665	4440		100% Be
M5	.500	.008		1830	3660		100% Be
M6	.500	.008	500°F AFTER 10 MINUTES AT 500°F	1145	2290	MAX. 2430 AVE. 2358 MIN. 2290	100% C
M7	.500	.008		1180	2360		100% C
M8	.500	.008		1158	2316		100% C
M9	.500	.008		1198	2396		100% C
M10	.500	.007		1215	2430		100% C
M11	.500	.007	650°F AFTER 10 MINUTES AT 650°F	733	1466	MAX. 1630 AVE. 1569 MIN. 1466	100% C
M12	.500	.007		807	1614		100% C
M13	.500	.008		755	1510		100% C
M14	.500	.008		812	1624		100% C
M15	.500	.007		815	1630		100% C
M16	.500	.003	800°F AFTER 10 MINUTES AT 800°F	510	1020	MAX. 1156 AVE. 1048 MIN. 850	100% C
M17	.500	.008		425	850		100% C
M18	.500	.008		544	1088		100% C
M19	.500	.007		562	1124		100% C
M20	.500	.007		578	1156		100% C

TABLE NUMBER 7

TEST RESULTS ~ BONDING METHOD "M" ~ AEROBOND 430 ADHESIVE

(SPECIMEN M5 NOT INCLUDED IN AVERAGE FOR ROOM TEMP. GROUP)

MAC 1008 (REV. 26 JAN. 59)
PAGE 1 OF 3

TEST REQUEST

TITLE Ablation Shield Development Testing: Adhesive Evaluation and Elevated Temperature Properties

LABORATORY OR DEPT. RESPONSIBLE FOR TEST 253 ~~252~~ (RKD)	MODEL 133N
TEST PARTS ON IBM ☐; ON TPL NO. PRODUCTION PARTS FOR TEST NOT REQUIRED ☐	APL/EPI

WORK REQUESTED

OBJECTIVE (GIVE PURPOSE OF TEST, WORK AND DATA REQUIRED INCLUDING SERVICE HISTORY AND BACKGROUND INFORMATION) Rev 6 ADDS ACTUALS 10-18-62 CMZ

1.0 PURPOSE: The purpose of this phase of the ablation shield development program is to evaluate several high temperature adhesives and determine their short-time elevated temperature shear properties. Rev. "C" Change in TR No. R. Winterton AEP 10-16-61 Refer MAC Rpt 8400 - Mark II Spacecraft Master Test Program & Schedule, Sect. 5.3.

2.0 MATERIALS:
(a) HT-424 Film, .015" thick, 0.13-0.14 lbs./ft.^{2} Bloomingdale Rubber Co., Aberdeen, Md.
(b) AF-107 Film, .015" thick, Minnesota Mining and Manufacturing Co., 6411 Randolph, Los Angeles 22, Calf.
(c) EC-1639 PRIMER, 20% solids, Minnesota Mining and Manufacturing Co., 6411 Randolph, Los Angelew 22, Calf.
(d) AEROBOND 430 , .015" thick, Adhesive Engineering Co., 1411 Industrial Road, San Carlos, Calf.

NOTES: (a) All of the above adhesives shall not be more than 30 days old at time of use.
(b) The Adhesives shall be stored in their original shipping container under the following conditions: 1. HT-424 : 0°F; (2) AF-107 and EC-1639 : 35-40°F; (3) AEROBOND 430 : 0°F .

3.0 FINGER PANELS FOR SHEAR TESTS: The finger panels shall be Brush Beryllium Co. QMV-200A press sintered block Beryllium machined (RMS-63) to the dimensions shown in Fig. 1 of P.S. 21330. Rolled sheet stock shall not be used. Rev. "D" Changes heating rate, curing method and adhesive. No change in estimate. TBR 11-15-61

4.0 SURFACE PREPARATION OF BERYLLIUM: Rev D Approved - No Schedule or Budget Change

(a) The Beryllium surface shall be prepared in accordance with the optimum procedure developed under T.R. 052-051.03.01. Method E was selected.
(b) At completion of the cleaning procedure, test panels shall be wrapped in wax-free brown Kraft paper.

REFERENCES OR ENCLOSURES
*See page 2
Rev. B. No Change in Estimate TBR 11-16-61

MATERIALS COST = [redacted] 5000

REV. "E" REVISES CHARGE NO [illegible]

4.0 (Continued)

(c) All bonding shall be performed within 24 hours after completion of the cleaning operation.

5.0 TEST CONDITIONS:

(a) For each adhesive, lap shear finger panels shall be prepared in a quantity sufficient to provide a minimum of 5 specimens for each test condition. (b) Test conditions are: (1) Room temperature, (2) 500°F after 10 minutes soak at 500°F, (3) 650°F after 10 minute soak at 650°F and (4) 800°F after 10 minute soak at 800°F. All the above temperatures are ± 10°F. In all cases, the bond line shall be at test temperature, as determined by proper instrumentation, for 10 minutes before testing starts. Load shall be applied at a rate of 1200-1400 psi per minute. The rate of heating the specimen shall be 100°F/minute.

6.0 BONDING PROCEDURE:

(a) GENERAL: The age and storage requirements of Section 2.0 shall be followed. The amount of adhesive necessary for a set of panels shall be removed from the roll and the roll returned to refrigeration. The adhesive to be used for the set of panels shall be wrapped in cellophane or Mylar and allowed to return to room temperature (approx. 2 hours) before being placed in the bond joint. All cure temperatures are based on bond line temperature, as determined by proper instrumentation. All panels shall be cooled to 150°F, or less, before pressure is removed. The 30 psi cure pressure shall be on the bond joint when increase in temperature is started.

(b) CURING OF HT-424:

METHOD 1: Cure at 30 psi, raise to 330 ± 5°F in 60 minutes and hold at 330 ± 5°F for 120 minutes.

METHOD 2: Cure at 30 psi, raise to 330 ± 5°F in 60 minutes and hold at 330 ± 5°F for 60 minutes, then raise temperature to 400 ± 5°F and hold for 60 minutes.

(c) CURING OF AF 107-EC 1639:

METHOD 1: Spray or brush on EC 1639 (thinner is MEK) to a film thickness of 0.0005. Air dry for 30 minutes followed by force dry of 30 minutes at 325° ± 5°F. Place adhesive in bond joint. Apply cure pressure of 30 psi. Slowly raise temperature to 350° ± 5°F at rate of 3°F/minutes (90 minutes elapsed time). Hold at 350 ± 5°F for 60 minutes.

METHOD 2: Apply primer as above. Slowly raise temperature to 245° ± 5°F at rate of 3°F/minute (55 minutes elapsed time); raise to 290° ± 5°F at rate of 1/2°F/minute (90 minutes elapsed time); raise to 350°F at rate of 3°F/minute (20 minutes elapsed time) and hold for 60 minutes.

* Applicable to IRAP unless final report is classified. Complete Report Summary Sheet, MAC 1008RS, per Engineering Procedure 3-23. Route report and Summary Sheet per Engineering Procedure 3-23.

TEST REQUEST

6.0 (Continued)

(d) CURING OF AEROBOND 430

METHOD 1: Using cure pressure of 30 psi raise temperature to 330°F ± 5°F in 20 minutes and hold at 330° ± 5°F for 60 minutes.

METHOD 2: Using cure pressure of 30 psi, raise temperature to 330° ± 5°F at rate of 5°F/minute (50 minutes elapsed time) and hold at 330° ± 5°F for 60 minutes.

7.0 REPORT:

(a) The report shall include the following information:

(1) Failing load,
(2) Failing stress level,
(3) Nature of failure and,
(4) Stress vs. Temp. curves for each adhesive.

8.0 SAFETY: All cutting, machining, grit blasting, etc. of Beryllium shall be performed under the cognizance and surveillance of Tom Linck, Safety Dept.

Advance notice must be given in order to have adhesives available.

ADHESIVE	TIME REQUIRED TO DELIVER - DAYS
HT-424	4
AF-107	7
EC 1639	7
AEROBOND 430	14